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Rock Island Arsenal Laboratory



TECHNICAL REPORT

HEAT RESISTANT CONVERSION COATINGS FOR STEEL
(Interim Report)

By

Linden H. Wagner

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**Rock Island Arsenal
Rock Island, Illinois**

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ABSTRACT

Zinc and cadmium oxides were dissolved in 75% phosphoric acid and used separately as stock phosphatizing solutions. They were diluted in the ratio of 1 to 4 and 1 to 8, by volume, or 1 to 15 by weight.. Steel wool was dissolved therein to raise the iron content. Zinc and cadmium oxides were made into a slurry with water, converted to the nitrates and added as accelerators. Steel panels were processed in the baths from 30 to 60 minutes at 190 - 195°F to obtain conversion coatings. The panels were divided into two groups. One group was used as the control, the other was heated in an oven at 300°F to determine heat resistance at that temperature. Both groups were evaluated by salt spray tests for their resistance to corrosion. It was found that cadmium coatings applied to the panels in the processing solutions, in the absence of iron and an accelerator, appeared to afford the best resistance to heat.

RECOMMENDATIONS

It is recommended that (1) complex crystalline coatings applied to steel in modified phosphatizing solutions should be further investigated to determine their resistance to heat and corrosion. (2) That other commonly used elements be investigated in the process solution for the possibility of forming complex crystalline coatings exhibiting thermal stability.

HEAT RESISTANT CONVERSION COATINGS FOR STEEL

CONTENTS

	<u>Page No.</u>
Object	1
Introduction	1
Procedure and Results	2
Materials	2
Zinc Phosphatizing Solutions	2
Cadmium Phosphatizing Solutions	4
Discussion	5
Literature References	7
Distribution	8

HEAT RESISTANT CONVERSION COATINGS FOR STEEL

OBJECT

To investigate heat resistant materials for their applicability to steel items, and to determine their corrosion resistance in environmental exposures.

INTRODUCTION

The treatment of iron or steel for preventing oxidation or rusting (1,2) has been used by the Department of Defense for years to maintain materiel in serviceable condition during periods of storage prior to issuance to the using unit. One of the processes used in the treatment of iron and steel is to immerse the item in a phosphatizing bath containing phosphates of iron and zinc, or iron and manganese, and an accelerator.(8) The applied coating is called a conversion coating because part of the basis metal is used in forming the complex crystalline coating.(3) Since the basis metal must form a part of the complex crystalline coating, iron must be in combination with the metallic elements supplied by the bath. Many studies have been made on the complexity of the coating and its behavior under environmental exposure. In past work conducted on the above coatings, it was found that they lose water of hydration when heated in an oven with subsequent reduction in resistance to corrosion.(4,5) Work at this Arsenal disclosed: "Zinc phosphate coatings formed at 200°F lose about 50% of the water of hydration when heated to 200°F in air. Seventy-five percent of the water is lost at 250°F and 98% at 450°F."

"Manganese phosphate coatings do not lose their water of hydration until a temperature of 250°F is reached. Water is then lost fairly linearly up to 300°F where there is a retention of approximately 10%."

Bearing the above facts in mind, this investigation was undertaken to determine the possibility of forming a complex crystalline coating, such as the phosphate-zinc-iron complex, by replacement or substitution of any or all of the commonly used elements and to comparatively evaluate the coatings for heat resistance. Since the loss of water by hydration from the crystalline structure results in decreased corrosion resistance, it was thought that an improvement in the coating could be effected by either removing the water of hydration or by controlling the amount in the conversion coating.

PROCEDURE AND RESULTS

Materials. Test panels, size 1 x 2 x 1/8 inches, with a 1/16 inch hole near the center of one edge, were fabricated from SAE 1020 steel listed in Federal Standard No. 66b. The panels were vapor degreased in trichloroethylene and grit blasted to renew the surface. Panels were processed from 30 to 45 minutes in the phosphatizing bath. They were then removed, rinsed in warm water, dried in a stream of air and divided into two groups. One group was placed in a forced draft oven at 300°F for two hours. The other group was used as controls. Each of the groups was placed in a salt spray (fog) cabinet operated in accordance with Method 811.1 of Fed. Test Method Standard No. 151 and comparatively evaluated.

The following processing solutions were prepared in an endeavor to develop a heat and corrosion resistant conversion coating:

Silicic Acid - Zinc - Iron Phosphatizing Solution.

The first solution was prepared by mixing silicic acid in reagent grade 75% phosphoric acid, so that the solution would hold as much silicic acid as possible. One hundred eighty grams of zinc oxide was dissolved in 500 ml. of 75% phosphoric acid in a one liter flask. The flask was made up to the mark with the phosphoric acid solution saturated with silicic acid. This stock solution was then diluted by volume in the ratio of one to four. Vapor degreased steel wool was added to the solution to build up the iron content and to decrease the free acidity. Twenty grams of zinc oxide was made into a slurry with distilled water. It was converted to zinc-nitrate with concentrated nitric acid. This solution was then added to the above. The following analysis was noted:

Free Acid	19.8 point TA/	= 4
Total Acid	38.8 point /FA	
Iron	0.4 %	

Panels were processed at 190° - 195°F for 30 minutes. One group was heated in an oven at 300° for 2 hours. The other group was used as controls. After one hour exposure in the salt spray, the heated and the control panels were rusted. The control panels were less rusted than the heated panels. The heat resistance of the coating was not satisfactory.

Since the previous phosphatizing solution was high in free acid and total acid, it was decided to prepare a solution in the ratio of 1 to 7. The analysis was as follows:

Free Acid	6.3 point	TA/	
Total Acid	46.2 point	/FA	= 7
Iron	0.3 %		

Two grams of zinc oxide per 100 ml. of solution was dissolved in concentrated nitric acid and added to the phosphatizing bath. Analysis was again made and the following was noted:

Free Acid	12.7 point	TA/	
Total Acid	66.6 point	/FA	= 6
Iron	0.3 %		

Panels were processed at 195° - 197°F for 30 minutes. One group was heated in an oven as indicated. The other group was used as controls. In the salt spray test, the control panels failed after 4 hours. The heated panels failed in one hour. The heat resistance of the coating was not satisfactory.

A third phosphatizing bath was prepared by dilution as before. Two grams of zinc oxide for each 100 ml. of solution was converted to zinc nitrate and added. Steel wool was processed. The analysis was as follows:

Free Acid	32.0 point	TA/	
Total Acid	143.5 point	/FA	= 4.5
Iron	1.7 %		

Zinc oxide was added directly to the solution to reduce the free acidity. The solution was then diluted as before. The analysis was as follows:

Free Acid	5.8 point	TA/	
Total Acid	26.8 point	/FA	= 4.6
Iron	0.3 %		

Panels were then phosphated in the bath at 190°- 195°F. for 30 minutes. One group of panels was heated in the oven. A second group of panels was used as controls. In the salt spray test, the heated and the control panels rusted in one hour. The heated panels were more rusted than the controls. The heat resistance of the coating was not satisfactory.

NOTE: A point is equal to 1 ml. of 0.1N NaOH when titrating a ten ml. sample.

Cadmium Phosphatizing Solution.

Cadmium oxide was dissolved in 75% phosphoric acid and converted to the phosphates. The excess acid was then decanted. The salt was redissolved in a mixture of phosphoric acid and distilled water and made up to a volume of 2 liters. This solution was used as a stock solution. The first bath was made by diluting the stock solution in the ratio of 1 to 15 by weight. A total of 1600 ml. of solution was prepared. One thousand ml. of this solution was then warmed to about 150°F. Steel wool was placed in the bath to decrease the free acidity and to add iron to the bath. After a period of time, the excess steel wool was removed and the solution was analyzed.

Free Acid	9.1 point TA/	= 5
Total Acid	41.6 point /FA	
Iron	.45 %	
Cadmium	0.01 gm/ml.	

Steel panels were then processed in the bath for 30 minutes at 190° - 195°F. The panels ceased gassing after 15 minutes. The coated panels were divided into two groups. One group was heated in an oven at 300°F for two hours, removed and cooled. The other group was used as controls.

Both groups were placed in the salt spray test. The coatings failed after one hour in test. The rust on the control panels and the heated panels was about the same. The heat resistance of the coating was not satisfactory.

A second phosphatizing bath was prepared in the ratio of 1 to 15 by weight as previously outlined. One thousand ml. of this solution was used without the addition of iron and an accelerator. Fifteen grams of cadmium oxide was added to the solution to decrease the free-to-total acid strength and to increase the cadmium content. Analysis was as follows:

Free Acid	10.8 point TA/	= 2.75
Total Acid	28.6 point /FA	
Cadmium	0.01 gm/ml.	

Panels were processed in the bath at 195° to 198°F for 60 minutes. The panels were still gassing at the time of removal. The coated panels were divided into two groups and treated as before. After one hour in the salt spray test, each group was rusted, however, the heated panels afforded better resistance to corrosion than did the control panels. This coating showed signs of being heat resistant.

Since the panels were still gassing when removed from the processing solution, ammonium hydroxide was added to decrease the free acidity. The solution was analyzed as follows:

Free Acid	3.6 point	TA/	= 7
Total Acid	27.7 point	/FA	
Iron	0.05 %		
Cadmium	0.01 gm/ml.		

Panels were processed in the bath of 195°F for 45 minutes. The gassing ceased after 20 minutes. The panels were divided into two groups and tested as before. After one hour in the salt spray test, a light green corrosion product was noted in each group of panels. The rust was more extensive on both the heated and unheated panels than experienced in the previous solution not containing ammonium salts. The coating was not satisfactory in its resistance to heat.

Since the second phosphatizing bath did not contain an accelerator, a third phosphatizing bath was prepared from the stock solution in the ratio of 1 to 15 by weight. Fifty grams of cadmium nitrate was added as an accelerator. Analysis was made of the solution before and after the addition of the accelerator.

Free Acid	10.9 point	13.0 point
Total Acid	36.2 point	47.0 point
TA/ FA	= 3.6	3.6

The solution was stabilized by heating at 195° to 197°F for 30 minutes. Panels were processed in the solution for 30 minutes at the above temperature. Very little gassing of the panels was noted after three minutes immersion. The panels were divided into two groups and treated as before. After one hour in the salt spray test, the heated group of panels was more rusted than the control group. The heat resistance of the coating was not satisfactory.

DISCUSSION

In order to develop a heat and corrosion resistant conversion coating on steel, it was desirable to review what had been accomplished in the past. It was noted that zinc and manganese metals were most commonly used in the phosphatizing baths; however, these coatings were not satisfactory because they lose water of hydration when moderately heated, which decreases their resistance to corrosion. It was decided to replace these commonly used elements by others and to evaluate these coatings. Since many silicates contain water, which can only be driven off at high temperature, and is not present as water of crystallization of adsorption, (9)

it was thought that the molecular bonds holding the water of hydration in the complex crystalline structure might be more receptive to the silicic acid molecules and replace that of water or produce compounds which lose their water of hydration at a higher temperature.

The thought in using silicic acid was to form a phosphosilicic acid which could be introduced into the phosphatizing solution. In the formation of the conversion coating, it was thought that the salts of phosphosilicic acid might displace the water of hydration in the complex zinc-iron molecule and thus, render the coating more resistant to heat and corrosion.

The tests show that the salts of silicic acid in the phosphate solutions had little or no affect on the applied coatings. On diluting the stock solution with distilled water, the acidity was reduced in strength. The concentration of the silicic acid was likewise changed, however, neither change had any influence on the applied coating.

Since zinc is commonly used in phosphatizing solutions and cadmium is found in the same series of the periodic chart of the atoms, it was decided to substitute this element in place of zinc, and to evaluate these coatings. The results show that cadmium coatings applied in the absence of iron and an accelerator in the phosphatizing bath produced a coating that was more heat and corrosion resistant than that of the control coatings.

The addition of cadmium nitrate to the phosphatizing solution did several things. First, it increased the concentration of the element in the solution. Second, it decreased the free-to-total acid ratio. Third, it speeded up formation of the complex coating. The results show that the control panels lasted four hours in the salt spray test, however, the heated panels failed in one hour. The coating was not satisfactory.

Cadmium was determined in the phosphatizing solutions in the presence of iron by volumetric method.⁽⁷⁾

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HEAT RESISTANT CONVERSION COATINGS FOR STEEL (Interim Report), by Linden H. Wagner

RIA Lab. Rep. 63-3345, 15 Aug 63, 14 p. incl.

illus., tables, (DA Project No. 1-A-0-10501-B-010, AMC Code No. 5011.11.83800) Unclassified report.

Zinc and cadmium oxides were dissolved in 75% phosphoric acid and used separately as stock phosphatizing solutions. They were diluted in the ratio of 1 to 4 and 1 to 8 by volume, or 1 to 15 by weight. Steel wool was dissolved therein to raise the iron content. Zinc and cadmium oxides were made into a slurry with water, converted to the nitrates and added as accelerators. Steel panels were processed in the baths from 30 to 60 minutes at 190-195°F to obtain conversion coatings. The panels were divided into two groups. One group was used as the control, the other was heated in an oven at 300°F to determine heat resistance at that temperature. Both groups were evaluated by salt spray tests for their resistance to corrosion. It was found that cadmium coatings applied to the panels in the processing solutions, in the absence of iron and an accelerator, appeared to afford the best resistance to heat.

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